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PROJECT 325B

SUMMARY LETTER REPORT

PERIOD: August 16, 1972 to September 15, 1972

Submitted By: [redacted]
Project Manager

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Gant Charts

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1.0 General

1.0.1 Personnel

The Junior Research Associate for Chemical R & D has been hired. He is [redacted] who has B.S. and M.S. degrees from Ohio University. He will begin work on September 18. His assignments will be in adding depth and expertise to purification and synthesis areas.

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1.0.2 New Coating Area

Construction of the new coating facility is complete. Delay in occupancy has been incurred, however, due to some delay in furnishing and equipping the facility. Occupancy by no later than the end of September is currently expected.

1.0.3 Final Report

The final report for the previous contract period (May to June 1972) is complete. It will be delivered by the end of September.

1.1 Chemical R & D

1.1.1 Inhibition of Speed Decay

Ingredient-interaction decay studies on the 5/D8 system are underway. They include both air and oxygen-free (argon) environments, which parallel previous studies with 5/D7. These studies will supplement existing data which indicate that 5/D8 may be a superior system with respect to sensitivity, nonuniformities and shelf life.

The CBr_4 loss-rate from film has now been determined by measuring the infrared absorption band of CBr_4 at 650-660 cm^{-1}. The data indicate that 50% is lost within 5 hours. Overcoated (PVA) films show no loss after 70 hours. D-Log E curves of 5/D7 for different concentrations of CBr_4 have been generated for both DPO and RLD modes. These curves combined with loss-rate data confirm loss of CBr_4 as a major cause of rapid speed decay and explain in part the more rapid decay observed in the RLD

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mode as compared to DPO mode. These data do not completely account for speed decay, however; as was expected in view of the postulated chemical speed decay mechanism.

Attempts have been made to simulate observed decay of standard 5/D7 films by adjusting the CBr_4 concentration to measured loss-rates and then adding 4DMAP. In this way D-Log E curves in the DPO mode have been simulated for the shorter decay periods of 1 to 3 hours. The corresponding curves for the RLD mode are not in agreement, however. These studies are imcomplete but the results to date suggest that chemical speed decay is more complicated than originally postulated. That this is the case is not surprising. The complications may merely involve secondary reactions of 4DMAP, but may involve still other mechanisms.

1.1.1.1 General Screening of Chemical Inhibitors

The initial mass screening of peroxide/hydroperoxide decomposers is well underway. Screening employs Formula 5 and 5/D7 without overcoating in both DPO and RLD modes. Several concentration levels are evaluated over a 24 hour period and compared with standard 5 and 5/D7 decays. Five classes of compounds have been screened thus far. They are: 1) sulfides, 2) disulfides, 3) thiolsulfinates, 4) mercaptans (thiols) and 5) metal xanthates. In all, thirty-four (34) compounds were evaluated. Details will be discussed in the October Technical Report. For the present, several sulfides and mercapto derivatives appear promising; they show higher Dmax's (less loss of Dmax) and in several instances with lower fog levels than controls after comparable aging periods (3 and 24 hours).

1.1.1.2 Combined Chemical/Overcoating Methods

Formula 5/D8 and its relation to retardations of sensitivity spots was described last month. The current promise of superiority of 5/D8 over 5/D7 has necessitated further optimization studies with regard to the RLD mode. Optimum wavelength for development is found to be between 683 and 707 nm. In contrast, PVA-overcoated films show optimum development with 717 nm. This is the first indication that optimum wavelength for RLD may change as a result of the overcoat.

The improvements made with 5/D8 were extended to 5/D7. 4PO was replaced by N5 and ethyl acetate was added as co-solvent. As a result sensitivity spot growth is retarded for a period of

5 to 6 hours. Overall sensitometry is also improved particularly as a result of lower gamma.

Last month overcoated 5/D8 was reported to last 19 hours at 70° with no significant spot growth and no significant loss of DPO sensitivity. It is now found that after 27 hours a new type of random spot growth appears. Though difficult to describe, these spots are different in appearance and apparently do not grow from crystal centers. It may be a result of chemical reaction between the aqueous overcoat and the photosensitive layer. An immediate possible explanation is reaction of water with CBr_4 to produce acid. A second possibility is the diffusion of 4PO to the PVA layer in which it is more soluble. Such a layer, without the PVA topcoat might also permit distinction of decay phenomena associated with loss of CBr_4 (and inherent chemical reactions) from reactions with oxygen. Several possible interlayers are now being investigated. They are polyvinyl acetate and cellulose acetate-butyrate.

The effect of pH of the PVA overcoating solution is being studied with regard to sensitivity spot growth as well as overall properties. Preliminary results indicate significant differences in photographic properties and aging.

The optimum N-oxide concentration for an overcoated film differs from the unovercoated film. This finding is not surprising if there is a competitive reaction between oxygen and N-oxide (cf. Proposal, July, 1972, Section 2.1.1.6, page 29).

1.1.2 Special Purifications

Samples of photograde D260 have been subjected to further purifications in the presence of several promising peroxide decomposers (cf. 1.1.1.1). Photographic evaluations were incomplete at the time of this writing.

1.1.3 Synthesis Program

1.1.3.1 D260 and Analogue

The initial attempt to reduce D260-carbinol to D260 has failed. Several products were obtained but none were D260 and none showed significant photosensitivity.

1.1.3.2 Peroxide Decomposers

Seven dithioalkanes have been prepared for screening. They are:

$CH_3(CH_2)$-$S(CH_2)_n$-S-$(CH_2)_xCH_3$ where:

n = 2,3,4 and x = 9,11 and n = 2, x = 17.

O ethyl-S-tert-butylthiosulphenyl xanthate was also prepared.

Three new N-oxides have also been synthesized. They are related to 4PO but are more soluble in benzene. They will be evaluated for replacement of 4PO and N5 with particular regard to sensitivity spot growth with overcoated material.

1.1.4 Ingredient Purification and Supply Maintenance

All principal film ingredients are in adequate supply.

Improved synthesis and purification methods are being explored for D263. An adequate supply for film evaluation and decay studies is expected by the end of September.

1.1.5 Analtyical Studies

Only a small effort has been expended on continued analysis of fractions from the column chromatographic work. Little progress is being made and it appears that another approach to dye separation will be necessary. Possibilities are being investigated.

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		DOC Jul	Aug	Sep	Oct	Nov	Dec	EOC
1.1	Chemical R & D							
1.1.1	Inhibition of Speed Decay							
1.1.1.1	General Screening of Chemical Inhibitors							
1.1.1.2	Combined Chemical/Overcoating Methods							
1.1.2	Special Purifications							
1.1.3	Synthesis Program							
1.1.3.1	D260 and Analogue							
1.1.3.2	Peroxide Decomposers							
1.1.4	Ingredient Purification and Supply Maintenance							
1.1.5	Analytical Studies							

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1.2 Engineering

Progress has been made in all phases of the Engineering program. The heavy work load required to construct the new darkroom facilities and the required equipment has posed a severe manpower loading problem. Every effort has been made to maintain the forecast schedule.

1.2.1 Calibration and Maintenance

Calibration and maintenance of all 325 equipment has continued unabated.

1.2.2 New Darkroom Facilities

The design of the necessary facilities was completed on August 15. The darkroom areas themselves including the electrical and air-conditioning supplies has been completed. The equipment required including red lite stations, coating plates, and sensitometers is still continuing. It has proved difficult to build this large number of pieces of equipment on schedule. The red lite units are nearing completion with an anticipated 2 week additional time required for their completion. The sensitometers have been completed. The coating plates await some machining and additional parts as yet undelivered by the vendor. The hoods and other work space equipment are being completed at this time. It is anticipated that the new darkroom facilities will be operational within 2 weeks.

1.2.3 Shelf Life Study

One of the major problems in improving shelf life has been finding methods and materials for proper overcoating. Several new overcoating PVA materials have been investigated with some success. Different molecular weight materials and percent solutions have been tried to determine optimum overcoating conditions. All tests up to this point have been done by either draw bar or dip coating PVA. Without exception the dip coating is the superior method. Although great improvement has been made regarding spot growth, it still continues to be a minor problem limiting maximum shelf life studies. Various other tests are being conducted to determine if such effects as particles, contamination, or acidity has an effect on spot growth or shelf life. The experiments are just getting underway and no data is available at this time.

A crude spin coater was quickly constructed in order to determine the basic parameters for spin coating. Preliminary data will be generated on the spin coater to determine exactly what type of equipment should be purchased.

The search for additional PVA materials is continuing.

1.2.4 Red Lite and Filter Tests

This area of investigation was suspended for the last month in order to concentrate effort and manpower on the equipment construction for the new darkroom facilities. This work will be resumed as soon as the darkroom facilities are operational.

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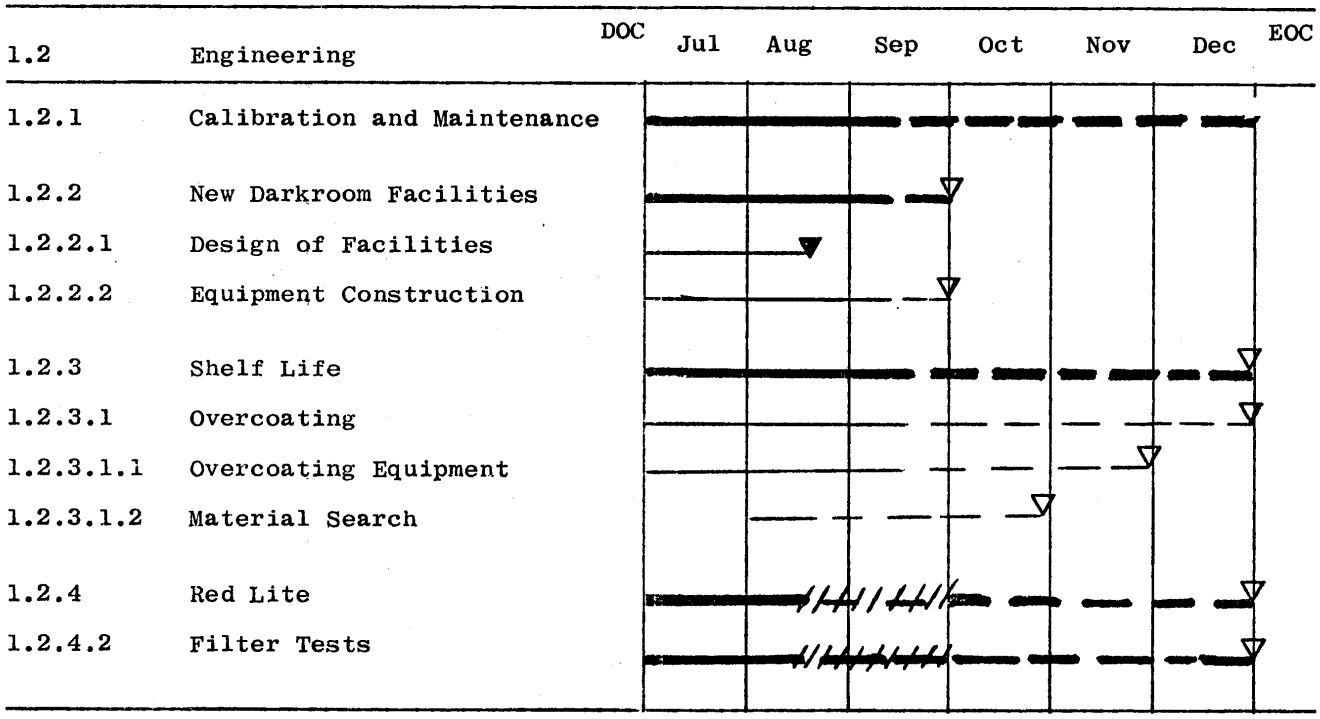

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Work Suspended

1.3 Perkin Elmer Interface and QC/QA

1.3.1 Perkin Elmer Interface

1.3.1.1 Plans for Perkin Elmer Program

The goals of Item 1 are being carried out under Item 2 of the 9 August program statement. Item 1 is being delayed until overcoated material is acceptable for shipping, which will be at least the end of October. As of the 15th September meeting the experimental plans are:

1) Complete red lite filter tests.

2) To determine the RLD rate dependence with the use of ND filters.

3) To run spectra of the unfixed shipped materials exposed and unexposed.

4) To see if raised temperature itself causes any development.

5) If possible, to make the first tests with a laser for development.

As soon as the QC/QA Interface lab area is up to speed, one of the first priority items will be to determine the effect of elevated temperatures on RLD of fresh material. Hopefully QC/QA will have the chance to determine what causes the loss in RLD speeds on shipped material before then. In any case, finding that speeds can be increased an order of magnitude by heating the platen during red lite development, holds some interesting possibilities. This may prove to be the first real impact of the Interface program on the shelf life problem. As of now the few bits of existing information are consistent with the belief that mobility of the chemicals may be important for RLD.

Perkin Elmer reports that RLD temperatures of 90°F, instead of room temperature, will yield roughly an additional 20X (total of 400X) amplification when using the red filter with a 650 nm cut-on. Perkin Elmer also established that, at room temperature,

switching of the RLD filter to one with a 695 nm cut-on yielded a much more desirable γ with approximately the same speed. The latest data indicate that raising the temperature with the longer wave cut-on filter will also yield increased speeds, although as of now they are roughly only a factor of six (6) instead of twenty (20). One of the "cleanest" looking samples seen to date used a red lite cut-on filter slightly short of the 695 nm at 96°F. The total amplification factor (the DPO control includes a factor of six) was 130. Observed during the 15 September trip were many developed samples that looked clean, had several toe steps and looked like printout samples. The sample was free of blotch and had a relatively low uniform fog outside the tablet area.

Perkin Elmer's various aging tests of both DPO and RLD, before and after exposure, and before and after development, have shown the current importance of these parameters for quality control. These results also fit in with the theory of reactivity associated with the dye's physical properties, such as mobility in a viscous polymer media and surface area/ weight ratio. A more detailed explanation of the results and conclusions will appear in the October Technical Report and Perkin Elmer's progress reports.

1.3.1.2 Liaison (Meetings)

Meetings with the consultants have taken place on July 7, and August 21 with the [redacted] and Perkin Elmer personnel. 25X1
A preliminary meeting had taken place with each of the consultants at Perkin Elmer prior to the July 7 meeting. The next meeting with the consultants is tentatively set for the 16th or 17th of October at [redacted] At the first meeting it 25X1
became apparent that the consultants had been given information on other film systems and some pre-325 data that rendered many of their prepared questions unappropriate. In order to prevent this, a list of all information sent to the consultants by Perkin Elmer will be sent to [redacted] will be kept 25X1
informed of all communications between Perkin Elmer and the consultants. The participation by the consultants in the second general meeting was much more relevant. All communication between [redacted] and the consultants must go through Perkin Elmer 25X1
although a sample of 4PO will be sent directly from [redacted] to 25X1
[redacted] upon approval of the Customer. 25X1

1.3.1.3 Film Shipments

Film Shipment 11 (first for this contract period) was sent 14 August. The twelfth shipment, which was really a

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supplement to Shipment 11, consisted of only 5 sheets instead of 15 and was sent the 28th of August. Shipment 13 was sent September 11 and consisted of 15 sheets.

1.3.2 QC/QA

1.3.2.1 Materials Inventory

Sufficient quantities for the larger coating effort have been maintained. Hand coatings are now being produced by nine technicians (six in Chemistry, two in Engineering, and one in QC/QA). The rate will increase further when the two remaining sets of equipment and three new lab rooms are completed and checked out by the end of September. The following photograde quantities were available as of 15 September.

	gm/ml Available	Handcoating Equivalent Amounts
D260	113 gm	2500
D7	208 gm	7300
CBr_4	850 gm 500 ml	1800
P.O.	33 gm 650 ml	4300
D8	47 gm	

1.3.2.2 Calibrations

The quality of Formula 5/D7 materials has been maintained at a barely sufficient level. The calibration of sensitometers and RLD units has been maintained on a weekly basis (minimum) without any problem. The new MacBeth TD-102 densitometer has been cross calibrated with original 325 and Perkin Elmer's TD-102 units. The original 325 densitometer will remain as the standard for now. It gives the lowest blue filter densities and γ of all three. It therefore is the harshest judge because the fall-off in density is the most commonly used indicator of poor materials and aging films.

A regular QC check of the Mylar thickness is now being made. For two weeks the check was made daily to establish a pattern, and is now made once a week. The variations in thickness across the web as measured with a Permascope EC-3-Ta, Model A, show that the coating thickness of approximately 1/3 mil ±5%. This variation of 10% is sufficient to cause gross nonuniformities such as the blotch. Because tne Permascope is a hand operated device with a probe diameter of .03 inches, the valleys in the Mylar must be at least .3 inches wide in order to be sure of detection. A sample was sent out and measured on a Calimike and then used as a gauge for the Permascope. A relatively narrow valley, approximately 10 microns deep was read as only a 1 micron valley on the Permascope. Anything less than 3 microns will tend to get lost without considerable amount of repeated measurements.

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1.3	Perkin Elmer Interface and QC/QA	DOC Jul Aug Sep Oct Nov Dec EOC
1.3.1	Perkin Elmer Interface	
1.3.1.1	Test Planning for Perkin Elmer (RLD, Chamber, Redo's)	
1.3.1.2	Liaison (Meetings)	
1.3.1.3	Film Shipments	
1.3.2	QC/QA	

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PROBLEMS

1. Contractor delays in completion of new darkroom coating facility.

2. Securing best quality polyester base material.

PLANS FOR NEXT REPORTING PERIOD

1. Push for completion and qualification of darkroom coating facility.

2. Continue project work along forecast schedule.

FINANCIAL

Project is tracking within labor and materials budgets. See project tracking graphs.

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